
The Interlayer Collapse during Dehydration of Synthetic Na_{0.7}-Beidellite: A ²³Na Solid-State Magic-Angle Spinning NMR Study¹

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Abstract: The dehydration and migration of the interlayer cation of the synthetic beidellite Na_{0.7}Al_{4.7}Si_{7.3}O₂₀-(OH)₄·nH₂O, were studied with solid-state ²³Na and ²⁷Al MAS-NMR, heating stage XRD, and thermogravimetric analyses (TGA, DTA). The ²³Na MAS-NMR of Na-beidellite at 25°C displays a chemical shift of 0.2 ppm, which indicates a configuration comparable with that of Na⁺ in solution. Total dehydration proceeds reversibly in two temperature ranges. Four water molecules per Na⁺ are gradually removed from 25° to 85°C. As a result, the basal spacing decreases from 12.54 Å to 9.98 Å and the Na⁺ surrounded by the two remaining water molecules is relocated in the hexagonal cavities of the tetrahedral sheet. The chemical shift of 1.5 ppm exhibited after the first dehydration stage illustrates the increased influence of the tetrahedral sheet. The high local symmetry is maintained throughout the entire first dehydration stage. During the second dehydration, which proceeds in a narrow temperature range around 400°C, the remaining two water molecules are removed reversibly without any change of the basal spacing.

Key Words: Beidellite • Dehydration • Interlayer collapse • ²³Na MAS-NMR

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